Australian Government
Department of Industry, Science and Resources

National Measurement Institute

CIPM MRA

NATA
WORLD RECOGNISED ACCREDITATION

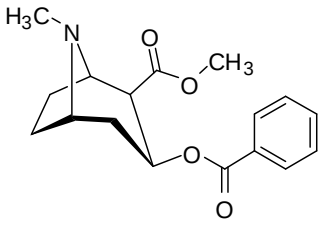
CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

NMIA D826d: Cocaine base

Report ID: D826d.2024.01 (Bottled 201124)

Chemical Formula: $C_{17}H_{21}NO_4$

Molecular Weight: 303.4 g/mol



Certified value

Batch No.	CAS No.	Purity (mass fraction)
19-D-02	**50-36-2**	**99.6 ± 0.4%**

The uncertainty has been calculated according to ISO Guide 35 and is stated at the 95% confidence limit (k = 2).

IUPAC name: Methyl (1R,2R,3S,5S)-3-(benzoyloxy)-8-methyl-8-azabicyclo[3.2.1]octane-2-carboxylate.

Expiration of certification: The property values are valid till 21 June 2027, three years from the date of certification provided the **unopened** material is handled and stored in accordance with the recommendations below. The material as issued in the unopened container and stored as recommended below should be suitable for use beyond this date, subject to confirmation of batch stability from the issuing body. The expiry date/shelf life does not apply to sample bottles that have been opened. In such cases it is recommended that the end-user conduct their own in-house stability trials.

Description: White powder prepared by synthesis, and certified for identity and purity by NMIA. Packaged in amber glass bottles with a septum and crimped aluminium cap.

Intended use: This certified reference material is suitable for use as a primary calibrator.

Instructions for use: Equilibrate the bottled material to room temperature before opening.

Recommended storage: When not in use this material should be stored at or below 25 ºC in a closed container in a dry, dark area.

Metrological traceability: The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. In the mass balance all impurities are quantified as a mass fraction and subtracted from 100%. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Stability: This material has demonstrated stability over a minimum period of three years. The measurement uncertainty at the 95% confidence interval includes a stability component which has been estimated from annual stability trials. The long-term stability of the compound in solution has not been examined.

Homogeneity assessment: The homogeneity of the material was assessed using purity assay by GC-FID on nine randomly selected 1-2 mg sub samples of the material. The material was judged to be sufficiently homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

Safety: Treat as a hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust. Refer to the provided safety data sheet.

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
24 June 2024

This report supersedes any issued prior to 24 June 2024.

NATA Accreditation No. 198 / Corporate Site No. 14214.

CIPM MRA notice: This certificate is consistent with the capabilities that are included in Appendix C of the MRA drawn up by the CIPM. Under the CIPM MRA, all participating institutes recognise the validity of each other's calibration and measurement certificates for the quantities, ranges and measurement uncertainties specified in the KCDB (for details see http://www.bipm.org/kcdb/). The "CIPM MRA Logo" and this statement attest only to the measurement(s) applied for determining the certified values on the certificate.

Legal notice: Neither NMIA as a representative of the Commonwealth of Australia, nor any person acting on NMIA's behalf, assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this document.

Characterisation Report:

The identity was confirmed by a range of spectroscopic techniques, NMR, IR and MS. The certified purity value was obtained by mass balance from a combination of traditional analytical techniques, including GC-FID, thermogravimetric analysis, Karl Fischer analysis and ^{1}H NMR spectroscopy. The purity value is calculated as per Equation 1.

Purity = (100 % - I_{ORG}) x (100 % - I_{VOL} – I_{NVR}) Equation 1

I_{ORG} = Organic impurities of related structure, I_{VOL} = volatile impurities, I_{NVR} = non-volatile residue.

Supporting evidence is provided by qualitative elemental microanalysis and the quantitative ^{1}H NMR obtained using the one-proton multiplet at 5.5 ppm measured against a certified internal standard of maleic acid.

GC-FID:	Instrument:	Varian CP-3800 or Agilent 7890A
	Column:	VF-1MS, 30 m × 0.32 mm I.D. × 0.25 μm
	Program:	160 °C (1 min), 15 °C/min to 200 °C (10 min), 30 °C/min to 300 °C (3 min)
	Injector:	200 °C
	Detector Temp:	320 °C
	Carrier:	Helium
	Split ratio:	20/1
	Relative mass fraction of the main component:	
	Initial analysis:	Mean = 100.0%, s = 0.002% (9 sub samples in duplicate, November 2019)
	Re-analysis:	Mean = 100.0%, s = 0.003% (5 sub samples in duplicate, July 2022)
	Re-analysis:	Mean = 99.9%, s = 0.015% (5 sub samples in duplicate, June 2024)
Karl Fischer analysis:		Moisture content ≤ 0.2% mass fraction (November 2019, June 2022 and June 2024)
Thermogravimetric analysis:		The volatile content could not be determined because of the inherent volatility of the material and non-volatile residue < 0.2% mass fraction (November 2019)
QNMR:	Instrument:	Bruker Avance-III-500
	Field strength:	500 MHz
	Solvent:	D_2O + DCl (4.79 ppm)
	Internal standard:	Maleic acid (98.8% mass fraction)
	Initial analysis:	Mean (5.5 ppm) = 100.3%, s = 0.2% (5 sub samples, November 2019)

Spectroscopic and other characterisation data

GC-MS:	Instrument:	Agilent 6890/5973
	Column:	DB-5MS, 30 m x 0.25 mm I.D. x 0.25 μm
	Program:	60 °C (1 min), 10 °C/min to 300 °C (3 min)
	Injector:	250 °C
	Split ratio:	20/1
	Transfer line temp:	280 °C
	Carrier:	Helium
	Scan range:	50-550 *m/z*
	The retention time of the parent compound is reported with the major peaks in the mass spectra. The latter are reported as mass/charge ratios and (in brackets) as a percentage relative to the base peak.	
	Parent (20.4 min):	303 (M^+, 24), 272 (10), 198 (13), 182 (100), 122 (11), 105 (38), 94 (38), 82 (95), 77 (33), 42 (18) *m/z*
TLC:	Conditions:	Kieselgel 60F_{254}. Methanol/conc NH_3 (200:3) Single spot observed, R_f = 0.7 Visualization with UV light (254 nm)
IR:	Instrument:	BioRad FTS3000MX FT-IR
	Range:	4000-400 cm^{-1}, KBr powder
	Peaks:	2977, 2947, 2885, 2852, 2803, 1737, 1709, 1450, 1317, 1279, 1229, 1112, 1038, 776, 714 cm^{-1}
^{1}H NMR:	Instrument:	Bruker Avance III-500
	Field strength:	500 MHz
	Solvent:	MeOH-d_4 (3.31 ppm)
	Spectral data:	δ 1.76 (2H, m), 1.90 (1H, m), 2.12-2.27 (2H, m), 2.21 (3H, s), 2.36 (1H, ddd, *J* = 2.8, 11.8, 11.8 Hz), 3.10 (1H, m), 3.29 (1H, m), 3.58 (1H, m), 3.69 (3H, s), 5.25 (1H, ddd, *J* = 12.0, 6.0, 6.0 Hz), 7.45 (2H, m), 7.58 (1H, m), 7.98 (2H, d, *J* = 7.1 Hz) ppm
	n-Hexane estimated at 0.2% mass fraction was observed in the ^{1}H NMR (November 2019)	
^{13}C NMR:	Instrument:	Bruker Avance III-500
	Field strength:	126 MHz
	Solvent:	MeOH-d_4 (49 ppm)
	Spectral data:	δ 25.9, 26.3, 36.4, 41.3, 51.2, 52.0, 62.9, 66.0, 68.2, 129.5, 130.6, 131.5, 134.3, 167.4, 172.4 ppm
Melting point:		98-99 °C
Microanalysis:	Found:	C = 67.7%; H = 6.9%; N = 4.6% (December 2019)
	Calculated:	C = 67.3%; H = 7.0%; N = 4.6% (Calculated for $C_{17}H_{21}NO_4$)